Determination of the Saturation Water Content of Protein Plastics

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FOR the correlation of chemical modification of proteins with changes in water absorption which they produce in the molded plastics the author has found it desirable to use the maximum water content of the plastic rather than water absorption values in which the rate of penetration is also a factor. A relatively short-time method has been developed which will determine the extent of water absorption of experimental protein plastics on immersion.

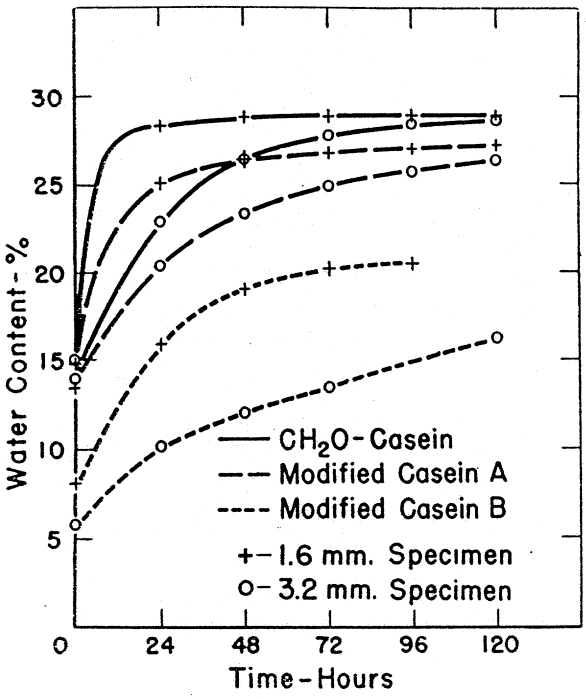

Figure 1. Water Uptake of Plastic Disks

The A.S.T.M. standard test for water absorption of plastics (*1*) is a "procedure for determining the relative rate of absorption of water by plastics when immersed". It was clearly shown in the work upon which the method is based (*2*) that the initial rate of absorption by different plastics is not necessarily correlated with the final equilibrium water absorption. For plastics of high water absorption, such as protein plastics, the A.S.T.M. method is especially limited, since the 24-hour initial conditioning at 50° C. does not bring the specimens to dryness or to any definite moisture content.

As in the previous work (*2*), preliminary experiments showed that the A.S.T.M. standard disk, 0.125 inch (3.2 mm.) thick and 2 inches (5.08 cm.) in diameter, did not reach saturation in several weeks. At the suggestion of R. W. Jackson, experiments were made with disks 1/16 inch thick in the hope that the water uptake of the thinner disks would reach saturation in a reasonable time. The thinner disks approached equilibrium much more rapidly than the standard disks, both on immersion and drying (Figure 1). The final approach to saturation and to dryness was essentially asymptotic. It was possible, however, to establish limits for the change in weight in 24 hours during sorption and drying, below which the disks could be considered to have reached an approximate saturated and dry weight. The author was thus able to obtain an "approximate saturation moisture content" in a reasonable time. Numerous experiments showed that for hot-molded protein plastics this value was between 95 and 100% of the saturation water content obtained after prolonged immersion and drying. Some samples of commercial casein plastics, which had been hardened with formaldehyde after extrusion, did not approach equilibrium in asymptotic fashion. In such cases, the approximate saturation moisture content determined by this procedure is an arbitrary value, since the saturation water content is uncertain.

PROCEDURE

PREPARATION OF SPECIMENS. Disks 2 inches (5.08 cm.) in diameter and 1/16 inch (1.6 ± 0.2 mm.) thick are used. They may be either compression-molded to the exact size or machined from bars or sheets of the finished plastic. If the bars or sheets are too narrow to be cut into 2-inch (5.08-cm.) disks, narrow strips may be used if they are 1.6 ± 0.2 mm. thick and their area is about that of the circular disk.

IMMERSION. The specimens are weighed only to the nearest milligram and placed in glass or wire racks in a container of distilled water, which is kept in a water bath maintained at 25° ± 0.5° C. They must be completely immersed, supported vertically, and separated from each other. At 24-hour intervals the specimens are removed, dried with a dry cloth, weighed rapidly only to the nearest milligram, and immediately returned to the water. (The loss by evaporation is 5.0 to 8.0 mg. per minute; consequently the weighings should preferably be made within 15 to 30 seconds.) When the gain in weight in a 24-hour period is less than 20 mg., or less than 10% of the gain in weight in the first 24 hours if the first 24-hour gain is less than 200 mg., the specimens are removed from the water and dried.

DRYING. To prevent disintegration by rapid expansion of entrapped moisture when dried at 105° C., the specimens are first partly dried by heating for 24 hours at 50° C. in a convection oven. They are removed from the 50° C. oven, weighed, and then heated at 105° C. If they are weighed 15 minutes after removal from the oven it is not necessary to cool them in a desiccator. Drying at 105° C. is continued until weighings made at 24-hour intervals show a loss of less than 20 mg., or less than 10% of the loss in weight during the first 24 hours of drying at 105° C. if this first 24-hour loss is less than 200 mg.

CALCULATION.

$$\text{Approximate saturation moisture content} = \frac{\text{wet weight} - \text{dry weight (105° C.)}}{\text{wet weight}} \times 100$$

The procedure has been in routine use in this laboratory for a year and a half. For most of the samples, the over-all time required for determination of the approximate saturation moisture content has been only 6 days. Making all measurements at 24-hour intervals effects an appreciable saving in the time of the operator, who can conveniently carry on other determinations concurrently. The procedure is applicable to protein plastics containing plasticizers except when the plasticizer is water-soluble. In these cases interpretation of the data in terms of the original plastic is difficult, but this is also true with the A.S.T.M. method.

Limited tests indicated that the method would be satisfactory for the experimental determination of saturation water content of cellulose acetate, urea-formaldehyde, and melamine-formaldehyde plastics. Phenolic resins and ethylcellulose, however, approach saturation slowly, and the thickness of the disk does not appear to be a major factor.

LITERATURE CITED

(1) Am. Soc. Testing Materials, Method D570–42.
(2) Kline, G. M., Martin, A. R., and Crouse, W. A., *Proc. Am. Soc. Testing Materials*, 40, 1273–82 (1940).